REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188

1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
	New Reprint	-

4. TITLE AND SUBTITLE

Controlling the electrostatic discharge ignition sensitivity of composite energetic materials using carbon nanotube additives

5a. CONTRACT NUMBER
W911NF-11-1-0439

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
611102

6. AUTHORS

Kade H. Poper, Eric S. Collins, Michelle L. Pantoya, Michael A. Daniels

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES

Texas Technical University
Box 41035
349 Admin Bldg
Lubbock, TX 79409 -1035

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)

U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSOR/MONITOR'S ACRONYM(S)
ARO

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
58857-EG.47

12. DISTRIBUTION AVAILIBILITY STATEMENT

Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT

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15. SUBJECT TERMS

energetic materials, carbon nanotubes, electrostatic discharge, aluminum, ignition, safety, combustion, solid fuels

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Michelle Pantoya
UU	UU	UU	UU		19b. TELEPHONE NUMBER 806-742-3563

Report Title

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REPORT DOCUMENTATION PAGE (SF298)
(Continuation Sheet)

Continuation for Block 13

ARO Report Number 58857.47-EG
Controlling the electrostatic discharge ignition se...

Block 13: Supplementary Note

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Journal of Electrostatics 72 (2014) 428 432

Contents lists available at ScienceDirect

Journal of Electrostatics

journal homepage: www.elsevier.com/locate/elstat

Controlling the electrostatic discharge ignition sensitivity of composite energetic materials using carbon nanotube additives

Kade H. Poper [a], Eric S. Collins [a], Michelle L. Pantoya [a, *], Michael A. Daniels [b]

[a] Department of Mechanical Engineering, Texas Tech University, Lubbock, TX 79409, United States
[b] Idaho National Laboratory, PO Box 1625, Idaho Falls, ID 83415, United States

ARTICLE INFO

Article history:
Received 12 June 2014
Accepted 28 July 2014
Available online 10 August 2014

Keywords:
Electrostatic discharge
Ignition
Aluminum
Thermites
Energetic materials

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Powder energetic materials are highly sensitive to electrostatic discharge (ESD) ignition. This study shows that small concentrations of carbon nanotubes (CNT) added to the highly reactive mixture of aluminum and copper oxide (Al + CuO) significantly reduces ESD ignition sensitivity. CNT act as a conduit for electric energy, bypassing energy buildup and desensitizing the mixture to ESD ignition. The lowest CNT concentration needed to desensitize ignition is 3.8 vol.% corresponding to percolation corresponding to an electrical conductivity of 0.04 S/cm. Conversely, added CNT increased Al + CuO thermal ignition sensitivity to a hot wire igniter.

1. Introduction

Powder composite energetic materials pose a particular threat to electrostatic discharge (ESD) ignition. These composites may be composed of solid fuel and oxidizer particles that produce exothermic reactions upon ignition. A common fuel is aluminum (Al) and can be combined with many different solid oxidizers including metal oxidizes like copper oxide (CuO) [1].

Weir et al. studied nine different composites and showed a correlation between measured electrical conductivity and ESD ignition sensitivity with Al + CuO ranking the most electrically conductive and most ESD ignition sensitive [2]. They also defined ESD ignition sensitivity as ignition below the threshold energy of 100 mJ but examined only micron scale particle composites. This was a first step toward identifying a measurable property of the reactants, such as electrical conductivity, and linking that property to ESD ignition sensitivity.

Weir et al. in Ref. [3] extended this correlation to nano scale particle composites and showed that aluminum combined with molybdenum trioxide (MoO_3) became significantly more ESD ignition sensitive as the Al particle size decreased. Aluminum particles inherently contain an alumina passivation shell that can range from 3 to 5 nm thick but is independent of particle size [4]. For this reason, as the aluminum particle size decreases, the inherent alumina concentration of the powder increases. Thus, there exists a trade off between the increased surface area to vol ume ratio of the nano scale particles that enhance diffusion controlled reactions versus the higher alumina concentration that can hinder energy propagation with properties that are more insulative. Weir et al. [3] showed that adding alumina to a micron Al + MoO_3 at an equivalent concentration to a nano Al + MoO_3 did not sensitize the mixture to ESD. But, for nano Al particles, ESD ignition sensitivity was increased by several orders of magnitude compared to micron Al + MoO_3 with equivalent alumina, which achieved no ignition. The interesting component of this study was that alumina that exists as a thin coating surrounding an aluminum core acts as a capacitive network and does not detract from ESD ignition, while an equivalent addition of alumina in bulk sizes on the order of 30 nm, prevents ESD ignition.

Electrostatic discharge ignition sensitivity as a function of the alumina passivation shell thickness was further examined in a study by Collins et al. [5]. Aluminum particles were synthesized with varied shell thicknesses and their response to ESD were measured in terms of ignition delay time. Thicker shells resulted in longer delay times and these results also correlated with measured electrical conductivity. Simulations were also performed using COMSOL multiphysics software and suggested the primary ignition mechanism was joule heating of the Al core as opposed to dielectric heating of the alumina shell.

The ESD ignition sensitivity of aluminum combined with poly tetrafluoroethylene (PTFE) was examined by Collin et al. [6]. They

* Corresponding author.
E-mail address: michelle.pantoya@ttu.edu (M.L. Pantoya).

http://dx.doi.org/10.1016/j.elstat.2014.07.004
0304-3886/

established the feasibility of manipulating electrical conductivity of the composite by using additives such as carbon nanotubes (CNT) to control the ESD ignition sensitivity. They showed with CNT ad ditives to Al + PTFE a narrow range of electrical conductivities (i.e., on the order of 0.0025 μS/cm) could be produced and resulted in ESD ignition [6]. They also showed Al + PTFE mixtures that were once insensitive became sensitive by controlling their electrical conductivity with the CNT additive [6].

All of these studies suggest that there exists an electrical con ductivity range that spurs ESD ignition. If a composite exhibits electrical conductivities that are either too low or too high, then the electric energy may either absorb or not absorb within the com posite enough to spur ignition or the electric energy may bypass the composite by channeling through an electrically conductive pathway such that energy buildup cannot happen.

The objective of this study is to examine variations in electrical conductivity and ESD ignition sensitivity for Al + CuO powders with varied CNT concentrations. A second objective is to determine how the CNT additive affects energy propagation once ignition is achieved. Percolation was determined from electrical conductivity measurements. Ignition sensitivity was evaluated using a standard ESD ignition apparatus reported in previous work [2,3,5,6]. Energy propagation was evaluated from flame speed tests using a high speed camera and analytical software. It is noted that the findings here are specific to micron scale Al + CuO particle composites but have implications towards ESD safety of other powder composite energetic materials.

2. Experimental

2.1. Materials

The multi walled carbon nanotubes (CNT) have an outer diam eter of 20 nm, an inner diameter of 3 nm, and a length varying from 0.1 to 10 μm. Aluminum (Al) powder has an average spherical particle diameter of 4.0 μm and copper oxide (CuO) powder has an average spherical diameter of 50 nm. All powders were procured from Alpha Aesar (Ward Hill, Massachusetts).

2.2. Mixing procedure

The masses for the fuel and oxide powders were calculated for a stoichiometric equivalence ratio. These proportions were combined with hexanes and sonicated for a total of one minute in ten second intervals. This cyclic program prevents damage to the alumina passivation shell during the mixing process. Sonication has been shown to be effective for producing homogeneous composites [7]. The composite was then poured into a glass dish and placed in a fume hood to evaporate and the dried composite was reclaimed for further testing.

2.3. Adding CNT

The concentration of CNT varied as a function of vol.% of Al + CuO. The masses corresponding to each vol.% were determined by first calculating the theoretical maximum density (TMD) for Al + CuO, and is 5.055 g/cc. The TMD was then used to calculate the CNT concentration for varied volumetric percentages. The volume percentages and their corresponding masses can be seen in Table 1. Before addition of CNTs all mixtures started with a mass of 350 mg of Al + CuO. Most experiments required less than 50 mg of powder, such that each sample preparation provided material for multiple tests. All experiments were run in triplicate to ensure repeatability of the measurements.

Table 1
Volumetric percent and mass of CNT added to Al + CuO and effective thermal conductivity of each mixture.

Set	Vol.% CNT	Mass of CNT (mg)	k_{eff} (W/mK)
1	0	0	19.5
2	0.5	1.8	21.3
3	0.75	2.7	22.2
4	1	3.5	23.0
5	1.25	4.45	24.0
6	1.5	5.4	24.9
7	2.25	8.1	27.6
8	3.08	10.9	30.4
9	3.8	13.6	33.1
10	4.6	16.3	35.8

Included in Table 1 is an effective thermal conductivity (k_{eff}) calculation based on a weighted average estimate for the thermal conductivities of each reactant. Values for the thermal conductivity, k, for each material are: k_{Al} 0.19 W/mK, k_{CNT} 3000 W/mK, k_{CuO} 72 W/mK [8]. It is noted that while the thermal conductivity of CNT is 5000 times that of Al, such small concentrations of CNT only slightly affect overall thermal conductivity.

2.4. Electrical conductivity measurements

A schematic diagram of the electrical conductivity setup is shown in Fig. 1. An acrylic channel was constructed to contain a constant mass of powder sample (i.e., 40 mg) such that the powder is positioned securely between two copper electrodes. The channel was placed inside a conductive shield to negate charges from the surroundings. A high resistance low conductance (HRLC) HR2 me ter from Alpha Labs (Salt Lake City, Utah) was connected to the two copper probes. For a more accurate measurement, the shielding container was held at ground potential by connecting it to the HRLC meter, which contains a high impedance amplifier.

The HRLC can measure a wide range or resistances varying from 1.0 Ω to 2.0 TΩ. The meter passes current through the sample at voltages below 2.0 V. The current that is passed through the sample decreases by a factor of ten for each resistance settings, of which

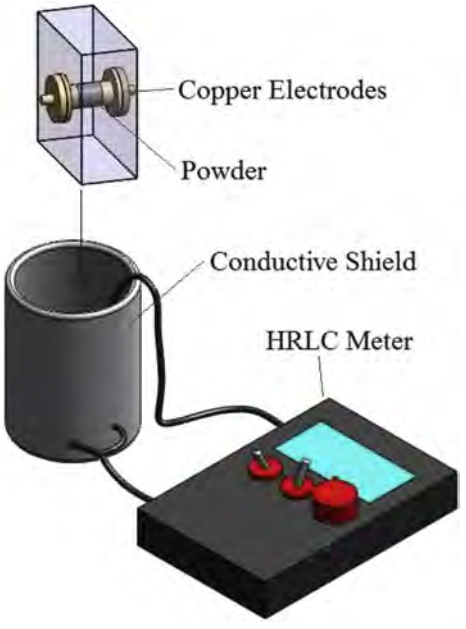

Fig. 1. Schematic of test setup for measuring electrical conductivity of powders.

there are nine. The current values can range from 0.1 mA to 1.0 pA for the 20.0 KΩ and 1999.9 GΩ resistances, respectively. Resistance was measured in KΩ and then inverted to determine conductance.

2.5. ESD sensitivity and minimum ignition energy tests

The ESD ignition apparatus was constructed by Franklin Applied Physics (Oaks, Pennsylvania) and tailored to evaluate powders. The apparatus was designed to model how a human body transfers an electric charge to other objects and is therefore referred to as a human body model ESD ignition tester [2]. The apparatus has a variable voltage supply that can range from 1 to 10 kV and charges a 0.002 μF capacitor. An electrode pin is connected to the capacitor and lowered towards the powder sample where electric energy is discharged from the pin to the sample. The same samples tested for conductance where also tested for the ESD ignition sensitivity. For ESD sensitivity, the 40 mg sample powder is contained in an insulated sample holder and ESD testing clearly resulted in ignition or no ignition. Minimum ignition energy (MIE) is tested using the same apparatus but instead of the highest voltage setting, subse quent voltages are used until a threshold is achieved where the indicated voltage no longer ignites the composite. The testing apparatus can be seen in Fig 2.

2.6. Flame speed tests

The setup for the flame speed measurements can be seen in Fig. 3. The powder sample is placed in a 10 cm long quartz tube of 3 mm inner diameter and 8 mm outer diameter. For these tests 300 mg powder sample is loaded into the tube and intermittently placed on a vibrating pedestal to remove large air voids and minimize density gradients. A Nichrome resistive heating wire is placed within the tube with a pinched center that acts as a point heat source that is in direct contact with the powder. The tube and wire are placed in a steel holder and also in a combustion chamber that has a viewing window. A Phantom v7 high speed camera is situated perpendicular to the direction of flame propagation. The Nichrome wire is attached to a variable voltage source and is turned to 15 V to reach a temperature for ignition. Each video is analyzed using a custom LabView program. The program tracks the front edge of the flame as it progresses through the flame tube. The difference in position from one frame to the next and the time

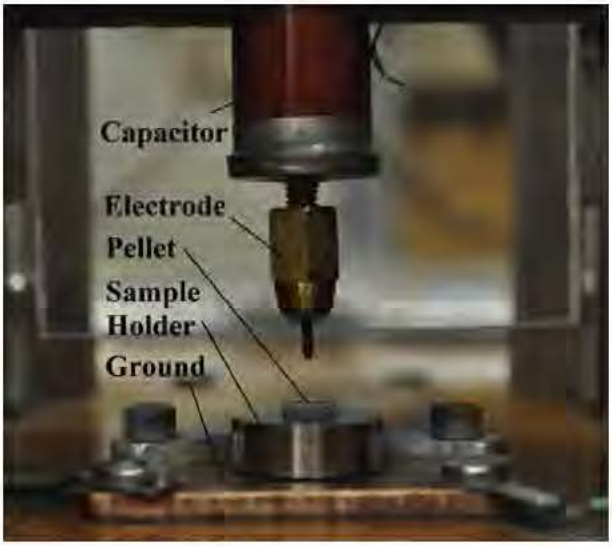

Fig. 2. Electrostatic discharge sensitivity/minimum ignition energy testing setup.

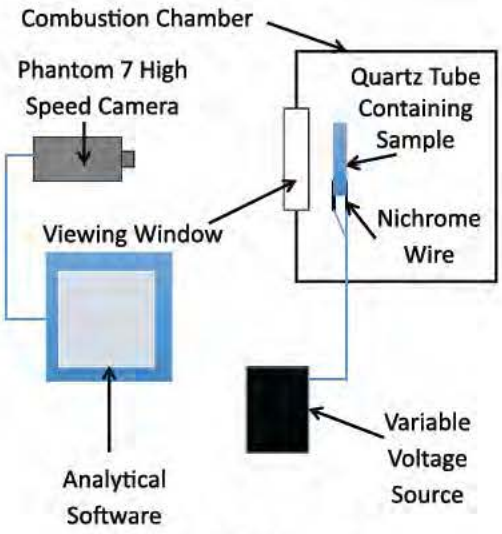

Fig. 3. Flame speed testing setup.

between each frame (0.06667 ms) results in flame speed. These experiments were run in triplicate to assess repeatability, the largest source of uncertainty in measuring flame speed.

3. Results

The CNT concentration ranged from 0.5 to 4.6 vol.% Al + CuO and Fig. 4 shows the results for both electrical conductivity and ESD ignition sensitivity tests. All ten mixtures were tested for conduc tivity, ESD sensitivity, and minimum ignition energy (MIE). For the ESD sensitivity a simple yes or no for ignition was given to each sample. In Fig. 4, the data with X symbols did not achieve ignition, all other samples ignited. An order of magnitude increase in con ductivity is seen between 3.08 and 3.8 vol.% CNT and this jump in electrical conductivity also corresponds to a repeatable change in ESD ignition sensitivity. Both of these behaviors suggest percola tion, after which the sample become insensitive to ESD. The average standard deviation for the conductivity measurements is 0.005%. Standard deviations represent uncertainty based on repeatability of triplicate experiments, the largest source of uncertainty in the measurements. Error bars in Fig. 4 data are therefore smaller than the data symbols presented.

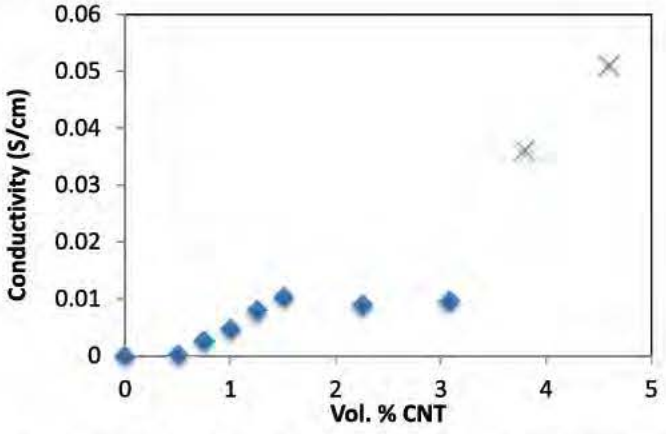

Fig. 4. Electrical conductivity of Al + CuO with added CNT. X symbols indicate samples that did NOT ignite during ESD testing.

Minimum ignition energy (MIE) is calculated using Eq. (1), where V is the applied voltage and C is the capacitance of the ESD apparatus.

$$\text{MIE} \quad \frac{1}{2}CV^2 \tag{1}$$

Fig. 5 shows MIE as a function of CNT concentration. As the conductivity of the samples rise with added CNT so does the MIE. The MIE for the samples containing 3.8 as well as 4.6 vol.% CNT are not shown because they were insensitive to the highest voltage setting (i.e., their MIE exceeded 100 mJ).

Scanning electron microscope (SEM) images in Fig. 6A–C shows powder morphology as a function of CNT concentration.

The base mixture containing no CNT and the samples with 3.08, and 3.8 vol.% CNT were tested for flame speeds to determine CNT effect on resistive wire ignition and energy propagation. Due to the use of micron aluminum particles, the base mixture did not ignite from the Nichrome wire. This was not a surprise because micron scale Al particles are inherently three orders of magnitude more insensitive to thermal ignition than nano scale Al particles [9]. Both the 3.08 and 3.8 vol.% CNT containing samples ignited when subjected to the point heat source and their corresponding flame speeds are 44.3 and 57.4 m/s (Table 2), respectively with 1% uncertainty based on repeatability of three separate flame speed tests for each sample.

4. Discussion

Fig. 3 indicates that a threshold for electrical conductivity may exist that significantly reduces ESD ignition sensitivity. The composites that show the highest electrical conductivity corresponding to 0.04 S/cm after percolation are desensitized to ESD stimuli. The electrical energy may be channeled through a CNT pathway and bypass energy buildup leading to ignition. The bypassing of the energy buildup can be seen in the minimum ignition energy (MIE) results shown in Fig. 4 because it takes greater MIE as the concentration CNT is increased. The MIE increases by 45 mJ from the base mixture without the CNT to the sample containing 3.08 vol.% CNT just before percolation. This jump in MIE is enough to render the sample safe to the maximum energy dissipated from a human being of 8.33 mJ [10] and far below the 100 mJ threshold identified in Ref. [3].

It is also interesting to note that for CNT concentrations between 1.5 and 3.08 vol.%, the electrical conductivity plateaus at approximately 0.005 S/cm but the MIE for these samples continuously

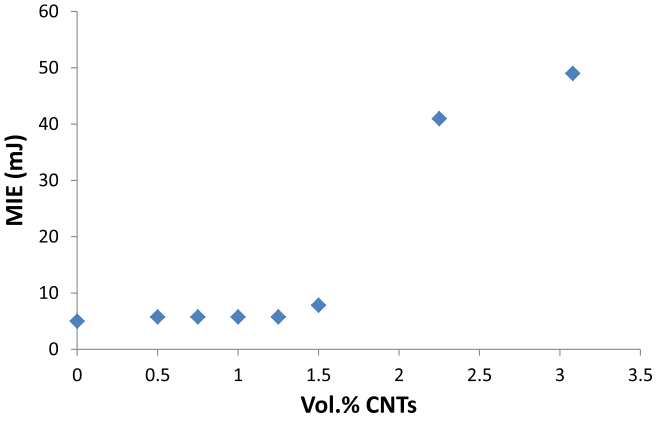

Fig. 5. Minimum ignition energy (MIE) of Al + CuO for varying vol.% CNT concentrations.

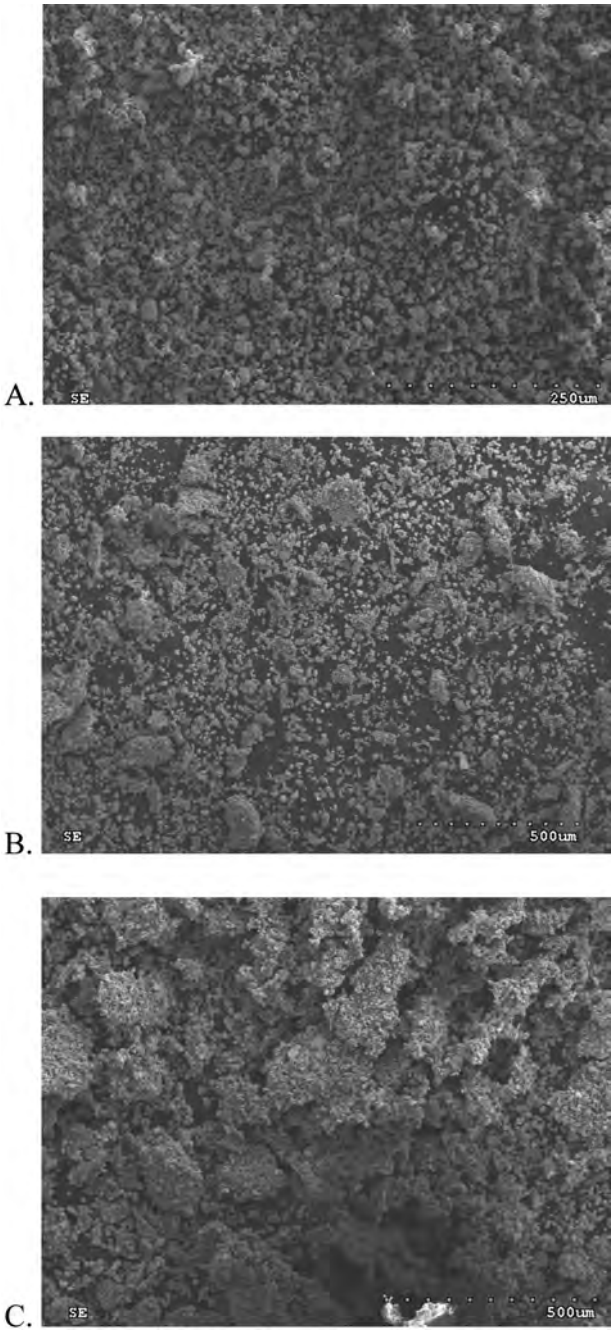

Fig. 6. SEM Images of A) 0 vol.% CNT, B) 3.08 vol.% CNT and C) 3.8 vol.% CNT.

increases and the samples remain sensitive to ESD stimuli. The SEM images shown in Fig. 6A–C help explain this behavior. Fig. 6A shows a homogenous mixture of Al + CuO. Fig. 6B is of Al + CuO + 3.08 vol.% CNT and shows that small agglomerations appear with the addition of CNT but their presence is scattered and sporadic. The SEM analysis reveals that inside these agglomerates are groups of CNT and because of their high conductivity they may attract the Al and CuO particles to form agglomerates. As CNT concentration increases, more agglomerations appear and the MIE increases. This becomes more apparent in Fig. 6C when the CNT concentration increased to 3.8 vol.%, agglomerations form a connected network throughout the sample. The overlapping of these agglomerates promotes connectivity of the CNT throughout the sample and gives the electric spark a path of least resistance when

Table 2
Flame speeds for samples ignited with the resistive Nichrome wire.

Mixture	Flame speed m/s
Al + CuO + 3.08%CNT	44.3
Al + CuO + 3.8%CNT	57.4

compared to that of the pre percolation samples. This new path results in a jump in electrical conductivity and samples become insensitive to ESD (Fig. 4); and, exhibit an increase in the minimum required energy to ignite the composite (Fig. 5).

In addition to desensitizing the sample to ESD stimuli, the CNT also sensitize (an otherwise thermally insensitive mixture) to the Nichrome wire as well as increase its flame speed. This increase in ignition sensitivity and energy propagation may be attributed to the large difference in thermal and electrical conductivity between the Al particles and CNT (0.19 compared with >3000 W/mK, respectively, Table 1). Assuming a constant heat flux from the Nichrome wire and constant temperature difference to achieve ignition among all samples tested, Fourier's Law [11] for conduction indicates that a region of thermal influence can be approximated as proportional to the thermal conductivity of the sample. As shown in Table 1, the thermal conductivity ranges from 19 to 35 W/mK such at higher CNT concentrations will provide a greater region of thermal influence.

Not only do CNT thermally sensitize the mixture but prior to percolation, CNT may also affect current flowing through the hot wire. The CNT may provide an additional path of least resistance for current to travel. Prior to percolation, as the current flows through the CNT and Nichrome wire, energy heats a larger region more effectively. In this way, a combination of electric and thermal en ergy promoted by CNT addition successfully extends the area of influence of the hot wire and helps to sensitize the mixture to thermal ignition and propagate energy.

It is also noted that the flame speeds measured for these micron scale mixtures (44.3 and 57.5 m/s, Table 2) are an order of magnitude less than the speed of nano scale Al + CuO composites examined under similar conditions (i.e., ~800 m/s) [12–15]. As Al particle size decreases, Al reactivity dramatically increases [16] and the overall trend of decreasing flame velocities with increasing Al particle size is readily seen for Al + CuO and similar formulations [17,18]. Because micron scale Al particles have a much smaller specific surface area, micron scale Al particles require higher tem peratures and longer duration at that temperature to achieve ignition and thus propagation. Along these lines, larger Al particles burn slower and produce significantly reduced flame speeds compared with nano scale particles. Flame speeds less than 100 m/ s are dominated by diffusive reaction mechanisms dependent on conduction as a mode of energy propagation. The increase in k_{eff} (Table 1) may contribute to the slight increase in flame speed with higher concentrations of CNT by enhancing conductive energy propagation.

5. Conclusion

This study shows that carbon nanotubes (CNT) added to a highly electrostatic ignition sensitive mixture of aluminum and copper oxide (Al + CuO) up to its percolation threshold desensitizes the mixture to electrostatic discharge (ESD) ignition. We have shown that an electrical conductivity threshold may exist corresponding to percolation that prevents ESD ignition by providing a conduit for electric energy to bypass the reactant powder. By bypassing the more resistive materials we have raised the minimum ignition energy above what the human body can discharge. The addition of CNT also made the composite sensitive to ignition from a resistive wire as well as promoted flame propagation. The increase in elec trical conductivity of the Al + CuO with CNT gives the current from the hot wire new routes to travel and successfully extends the hot wires region of thermal influence. Without CNT, ignition and propagation cannot be achieved using this resistive wire approach. These results are a first step towards significantly impacting future methods to control the ESD ignition sensitivity of powder energetic materials and enhancing their overall safety.

Acknowledgments

The authors M. Pantoya, E. Collins and K. Poper are grateful for support from the Army Research Office contract number W911NF 11 1 0439 and encouragement from our program manager, Dr. Ralph Anthenien. Idaho National Laboratory is also gratefully acknowledged for supporting this collaborative work with internal funds via the LDRD program.

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